

### **OLM04.3**

#### **Corrections/Modifications/Clarifications**

In 1999, EPA procured commercial laboratory services under OLM04.2 contracts. The scope of service consisted of the OLM04.2 Statement of Work (SOW) for the analysis of organic compounds found at Superfund sites. In early 2000, the CLP issued a modification (including technical changes) to the OLM04.2 SOW. This was outlined in the OLM04.2A Contract Modifications document. Specifically, a new SOW was not developed, but rather the scope of service became a combination of the OLM04.2 SOW and the OLM04.2A Contract Modifications document.

In March of 2003, EPA procured new commercial laboratory services under OLM04.3 contracts. Again, a new SOW was not specifically developed for this contract. The scope of service for the OLM04.3 contracts consists of the full length OLM04.2 SOW, the OLM04.2A Contract Modifications, and additional OLM04.3 modifications to both documents. Both OLM04.2A and OLM04.3 modifications can be found in this document.

#### **Exhibit C, Section 1.0 Volatiles Target Compound and Contract Required Quantitation Limits**

##### **OLM04.2A Modification Changes:**

All values under the column “Med. Soil ug/Kg” should be changed from 1200 to 1300. The reason for this change lies in the fact that the CLP rounding rule has been changed from that used in previous Statement of Work. The numbers in the “Med. Soil” column are derived from calculations based on the sample preparation protocol of the SOW. The calculated “nonrounded” value under this column is 1250 ug/Kg. Based upon the previous SOW rounding rule (if number to be retained is an even number preceding a 5, then number is to be unchanged), 1250 ug/Kg would be rounded to 1200 ug/Kg. The new rounding rule (as outlined in Exhibit B, Section 3.3.9, page B-33) stipulates that if the number to be dropped is 5 or greater, then the number retained should be increased by 1. Based upon this requirement, 1250 ug/Kg becomes 1300 ug/Kg.

*OLM04.3 Modification Changes: No changes are to be made to the above language.*

#### **Exhibit D - Volatiles Appendix B**

##### **Modified SW-846 Method 5035 for Volatiles in Low Level Soils**

##### **OLM04.2A Modification Changes:**

**Section 1.1 - The following statement should be added to the end of the paragraph.**

(It has been Superfund’s desire to make Method 5035 the predominant method for low level volatile soil analysis. However, due to technical uncertainties and logistic problems, the method has so far had limited use. AOC now believes that most of the technical and logistic issues have been resolved, at least on an interim basis, and therefore will be requesting the Regions to specify the use of the CLP Method 5035.)

*OLM04.3 Modification Changes: No changes to are to be made to the above language.*

##### **OLM04.2A Modification Changes:**

**Section 7.1.1 - Delete 7.1.1 as written and insert the following.**

Soil/sediment samples should be collected in the field in either field core sampling/storage containers (i.e. Encore™ or equivalent) or pre-prepared closed-system purge-and-trap sample vials as described in Section 9.3. If field core sampling containers are used, the field should send at least three containers per field sample containing approximately 5 g each, and at least one 60 mL sealed glass vial containing sample with minimum headspace. The Contractor shall transfer the contents of the field core sampling containers immediately upon receipt into the closed-system sample vial prepared as described in Section 9.3 below and record the date and time of transfer. If

pre-prepared closed-system purge-and-trap sample vials are used to collect samples in the field, the field should send at least two closed-system containers per field sample containing approximately 5 g each, and at least one 60 mL sealed glass vial containing sample with minimum headspace. The Contractor is then to proceed to Section 9.3.9 and determine final sample weight. If the minimum amount of containers have not been sent by the field, the Contractor is to immediately contact SMO for instructions. Note. If MS/MSD analysis are required for a particular sample, 2 additional field core sampling/storage containers or two additional closed-system purge-and-trap sample vials should be sent by the field. Contact SMO if insufficient sample for MS/MSD analysis has been sent by the field.

#### ***OLM04.3 Modification Changes:***

***The EPA Regions have been notified of the preferred means of collecting and delivering samples to CLP laboratories. The preferred mechanisms is collection into preweighed, non preserved (i.e. not containing sodium bisulfate preservative) closed-system purge-and-trap sample vials, or field core sampling/storage containers (i.e. Encore™ or equivalent). However, it has been made clear to the Regions that CLP laboratories will be able accommodate soils samples collected and shipped via numerous collection mechanisms (including methanol preservation, sodium bisulfate preserved closed-system vials, low headspace vials or jars, and coring sleeves).***

***If laboratories receive soil samples in pre-weighed, dry closed-system vials (no liquid inside the vial), the laboratory shall immediately place all sample vials (including any MS and MSD samples) in an isolated freezer (containing to organic standards or sample extracts) constantly held at a temperature colder than -7°C. The sample TR form or other field supplied documentation should contain information as to the sample weights, or at least to the tare weights of the vials. If no sample weight information or tare information is supplied, the laboratory is to immediately contact SMO for guidance. If tare weights only are supplied, the laboratory must before analysis, dry the vials, and then weight then to determine a vial plus sample weight. The laboratory shall subtract the vial tare weight from this weight to determine the net sample weight. The samples shall remain frozen until time of analysis, when they are to be taken from the freezer and allowed to come to ambient temperature before analysis. The samples shall then be placed upon the instrument autosampler for analysis. For the dry vials, the autosampler shall be programmed to introduce 10 mL of water via needle through the vial seal. The sealed cap shall not be removed at any time from the vials.***

***In some instances, the field will send samples that contain up to 5 mL of water, or 5 mL of water containing a sodium bisulfate preservative. These vials are also to be frozen up until the time of analysis. The laboratory shall place these samples in the freezer in a horizontal manner so that the vial will not burst upon freezing. The TR document shall include information as to whether these samples have been preserved with sodium bisulfate. If the samples have not been preserved, then the laboratory shall not preserve them. If the samples have been preserved, there is no need to analyze calibration standards that have also been preserved (i.e. there is no need to run calibration standards containing sodium bisulfate).***

***For samples received in the Encore™ type of device, the samples are to be immediately placed into tared, dry closed-system purge-and-trap vials, then re-weighed to obtain the final sample weight. The vials are then to be placed into a freezer. No sodium bisulfate preservative is to be added.***

***There may also be instances in which there are instructions on the TR that instructs the laboratory to preserve the sample in the laboratory. In this case, the lab shall add the preservative and analyze the samples as per the original instructions in the OLM04.2A modification instructions (original 7.1.1 modification). Care should be taken to first test a portion of sample in a sodium sulfate solution to check for excessive effervescence. If effervescing occurs, contact SMO for guidance.***

***Samples may also arrive as methanol preserved samples. These samples are to be considered to be medium level prepared samples, and are to be analyzed as per the original Statement of Work. These samples do not require freezing, but they shall be refrigerated at 4°C until time of analysis.***

**OLM04.2 Modification Changes:**

**Section 7.1.2 - Change wording to include the following.**

All samples received in closed-system purge-and-trap sample vials must be iced or refrigerated at 4°C (+/- 2°C) from time of collection until time of analysis. Any sample received in field core sampling/storage containers that are not immediately transferred to a closed-system purge-and-trap sample vial containing sodium bisulfate preservation solution must be placed in a freezing compartment and held at -12°C (+/- 2°C) until time of analysis (see Section 9.3).

**OLM04.3 Modification Changes:**

*All samples received in unpreserved closed-system purge-and-trap sample vials must be placed in a freezer contained at temperatures cooler than -7°C until time of analysis. Any sample received in field core sampling/storage containers shall immediately upon receipt at the laboratory transfer the sample from the sampling container to a dry, tared closed-system purge-and-trap-vial. The vial and sample are then to be weighed to determine the sample weight. The sample vial shall then be placed in a freezer contained at temperatures cooler than -7°C until time of analysis.*

**OLM04.2 Modification Changes:**

**Section 7.2.1 - Add the following language.**

The samples must be protected from light and refrigerated at 4°C (+/- 2°C) ( -12°C , +/- 2°C, if placed in freezing compartment as stated in 7.1.2) from the time of receipt until 60 days after delivery of a reconciled, complete sample data package to the Agency. After 60 days, the samples may be disposed of in a manner that complies with all applicable regulations.

**OLM04.3 Modification Changes:**

*Unused frozen samples must be kept frozen from the time of receipt at the laboratory until 60 days after delivery of a reconciled, complete sample data package to the Agency. After 60 days, the samples may be disposed of in a manner that complies with all applicable regulations.*

**OLM04.2 Modification Changes:**

**Section 9.3.1 - Add the following “Note” to the end of the paragraph.**

NOTE: There should be three field core sampling/storage containers for each field sample. The contents of two of the field core containers are to be processed using the steps outlined in 9.3.2 through 9.3.9. One of these prepared samples is then to be used as the primary sample, while the other is to be used as a backup sample, if necessary. The contents of the third field core container shall be transferred to a tared dry closed-system purge-and-trap container (i.e. no preservative solution or stirring bar is to be added), weighed according to 9.3.9, and then stored in a freezing compartment at -12°C (+/-2°C). This sample shall be used for the medium concentration level methanol extraction procedure as described in Exhibit D-VOA Section 10.1.4.2 if results of the original analysis indicate that medium level extraction is warranted.

**OLM04.3 Modification Changes:**

*Sampling guides are being delivered to Agency personnel detailing the number of samples required for analyses. Upon receipt from the laboratory, all soil sample containers requiring volatile analyses shall be frozen. This includes closed-system vials received from the sampler, closed-system vials generated by the laboratory, low headspace vials containing sample for moisture determination (or for analysis by the old Method 5030 protocol), and all QA samples.*

**OLM04.2A Modification Changes:****Section 9.3.8 - Change the language for “Note” to the following.**

NOTE: Soil samples that contain carbonate minerals may effervesce upon contact with the acidic preservative solution in the sample vial. Therefore, if samples are known or suspected to contain high levels of carbonates, a test sample (from the 60 mL glass vial) should be added to a clean vial and checked for effervescence. If a rapid or vigorous reaction occurs, the Contractor may discard the test sample and proceed with sample preparation by transferring the contents of each of the three field core sampling/storage containers into separate tared dry closed-system purge-and-trap vials (i.e. contains no preservative solution), two of which should contain stirring bars. Weigh the vials as per 9.3.9, and then transfer all three vials to a freezing compartment at -12°C (+/- 2°C). If analysis is to begin immediately on the sample, the contents of one of the field core sampling/storage containers may be transferred to a tared closed-system purge-and-trap vial containing 5 mL reagent water (no sodium bisulfate). The sample is then analyzed by the procedure starting at 9.3.9.

***OLM04.3 Modification Changes: No changes are to be made to the above language.***

**OLM04.2A Modification Changes:****Section 9.4.1 - Change the language to the following.**

Prior to sample purge, all soil/sediment samples must be allowed to warm to ambient temperature. For those samples that have been stored in freezing compartments and that will be analyzed by the low concentration level protocol, 5 mL of reagent water must be added to the vials without disturbing the hermetic seal of the sample vial (note: an additional 5 mL of reagent water will be added to the vial as per 9.4.2). Shake all vials containing aqueous solutions gently, to ensure that the contents move freely and that stirring will be effective. Place the sample vial in the instrument carousel according to the manufacturer’s instruction.

***OLM04.3 Modification Changes:***

***At time of analysis, the closed-system purge-and-trap vial must contain 10 mL of water. If the sample has been frozen without water, the laboratory purge-and-trap instrument may be capable of automatically adding 10 ml of water. If the instrument is preset to only deliver 5 mL of water, the laboratory will have to use a syringe and narrow bore needle to “inject” 5 mL of water before placing onto the instrument autosampler.***

**Additional OLM04.2A Modification Changes****Non-Method 5035 Modifications****Exhibit D-VOA**

**Section 10.1.3.7, Exhibit D, page D-30/VOA** stipulates that 10 uL of system monitoring compound spiking solution and 10 uL of internal standard spiking solution should be added to each 5 mL water sample analyzed. This requirement is based on manually operated purge-and-trap instrumentation. Purge and trap instrumentation is widely available in which internal standard and system monitoring compounds are automatically added to each sample. Some of this instrumentation may be set-up by the manufacturer to add only 1 uL of internal standard or system monitoring compounds. The 1 uL addition of standards will be allowed if the addition is done solely in an automated manner, and if the final concentration of standard in the 5 mL water sample remains the same as specified in the current SOW, i.e. 50 ug/L internal standard analyte and system monitoring compound concentration.

***OLM04.3 Modification Changes: No changes are to be made to the above language.***

**Section 10.1.4.1, Exhibit D, page D-31/VOA** stipulates that the Contractor (the laboratory) must determine whether a soil or sediment sample should be analyzed by the low or medium level protocols outlined in the SOW. Specifically, “it is the responsibility of the Contractor to analyze the sample at the correct level.”

This Section should be modified to indicate that if the Agency specifically requests the laboratory to analyze a sample only by the medium level protocol (i.e. methanol extraction technique), the laboratory is not obligated to perform the low level analysis. The request to the laboratory is to be made on the Traffic Report. After receiving a Traffic Report with this specific request, the laboratory is to confirm the request through SMO.

***OLM04.3 Modification Changes: No changes are to be made to the above language.***

#### **Technical Clarifications:**

There is some inconsistency in the SOW for the amount of sample to use when performing “medium level soil preparation” for volatiles. Section 10.1.5.3 of Exhibit D, page D-33/VOA instructs one to add 4 grams of soil sample to 10 mL of methanol at the laboratory, while Section 8.1.4 instructs one to add 5 grams of soil sample to 10 mL of methanol.

The 4 grams sample size is to be used when performing the medium level preparation in the laboratory, i.e. the laboratory has received a container of soil which is to be extracted with methanol in the laboratory. The 5 gram sample size is used when soil samples are preserved with methanol in the field. The Medium Soil Quantitation Limits in Section 1 of Exhibit C are based upon the “in laboratory” protocol using 4 grams of sample. Even though the “in field” protocol would present a lower Quantitation Limit (1000 ug/Kg), the required Quantitation Limit will remain at 1300 ug/Kg.

***OLM04.3 Modification Changes: No changes are to be made to the above language.***

There is also some confusion in defining SDG when preliminary results are involved. The SOW states in Section 4.2.2.1.1 Exhibit A page A-6 that all samples and sample fractions assigned to an SDG must have been scheduled under the same contractual turnaround time. This means that, for example, if 10 samples for volatile analysis were sent to a lab, and preliminary results were requested for one of the 10 samples, then two separate SDGs would have to be created; one SDG for the one preliminary results sample and one SDG for the remaining 9 samples. Because two SDGs have been created, then according to the SOW, a separate delivery package would have to be created for each SDG, and separate MS/MSD analysis would have to be analyzed for each SDG. Agency CLP users are being requested to use the preliminary results option judiciously. An attempt will be made to ensure that appropriate turnaround options are requested and that unnecessary QC and reporting packages are not performed. Under the new SOW, MS/MSD analysis is not required if the Agency requests that it not be performed. The lab is requested to make sure that SMO is contacted if there is any question or concern at all over assigning SDGs.

***OLM04.3 Modification Changes: No changes are to be made to the above language.***

#### ***Additional OLM04.3 Modification Changes:***

***Section 11.2.1.4 of the SOW details a formula used to calculate results of Medium Level Soil Analysis. This formula was developed without the consideration of moisture content in the soil. Even though, for CLP, a moisture correction is made to the results to convert from wet weight to dry weight, the actual volume of water in the sample that becomes miscible with the methanol solvent has not been accounted for. For example: If a 5 gram soil sample contains 10% moisture, the effective volume of liquid present in the sample extract after***

adding the method required 10 mLs of methanol now becomes 10.5 mLs (10 mLs methanol, 0.5 mLs water from the soil). If this effective volume is not taken into account inaccurate results of final data will occur, especially with samples containing high moisture content. Therefore, under this modification, the formula found in section 11.2.1.4 of the SOW shall be modified to the following:

$$\text{Concentration ug/Kg (dry weight basis)} = \frac{(A_x) (I_s) (AV_t) (1000) (DF)}{(A_{is}) (RRF) (V_a) (AW_s)}$$

Where,

$A_x$ ,  $I_s$ ,  $A_{is}$  are as given for water, Equation 5.

$RRF =$  Relative response factor from the ambient temperature purge of the calibration standard.

$V_a =$  Volume of the aliquot of the sample methanol extract (i.e., sample extract not including the methanol added to equal 100  $\mu\text{L}$ ) in microliters ( $\mu\text{L}$ ) added to reagent water for purging.

$$D = \frac{100 - \% \text{Moisture}}{100}$$

$DF =$  Dilution Factor. The dilution factor for analysis of soil/sediment samples for volatiles by the medium level method is defined as:

$$\frac{\mu\text{L most conc. extract used to make dilution} + \mu\text{L clean solvent}}{\mu\text{L most conc. extract used to make dilution}}$$

$AV_t =$  Adjusted total volume of the methanol extract plus soil water in milliliters (mL), determined by:

$$AV_t = V_t + \{Ws - [(Ws)(D)]\}$$

Where  $V_t$  = Total volume of the methanol extract in milliliters (mL). This volume is typically 10 mL, even though only 1 mL is transferred to the vial in Section 10.1.5.6.

$Ws$  = Weight of soil/sediment, in grams (g).

$AW_s =$  Adjusted weight of soil/sediment extracted, in grams (g), determined by:

$$AW_s = (Ws)(D)$$

OLM04.2 to OLM04.3  
Summary Of Changes

The OLM04.2 SOW document has been revised to OLM04.3 as identified in the Exhibit section(s) (and any other applicable sections within the OLM04.2) shown below. All changes identified in this document should be adhered to in conjunction of the OLM04.2 SOW as stipulated below.

Exhibit Section(s)	Revisions
Global	<p>Unless otherwise identified, all references to:</p> <ul style="list-style-type: none"> <li>USEPA Administrative Project Officer and USEPA Technical Project Officer (TPO) are changed to USEPA Regional CLP Project Officer (CLP PO).</li> <li>EMSL/LV are changed to QATS.</li> </ul>
Global	All references to "OLM04.2" are changed to "OLM04.3".
<p>Exhibit A: Section 4.2.2.1.1</p> <p>Exhibit G</p> <p>Each</p>	<p>A Case consists of one or more SDGs. An SDG is defined by the following, whichever is most frequent:</p> <ul style="list-style-type: none"> <li>Each Case of field samples received, or</li> <li>Each 20 field samples [excluding Performance Evaluation (PE) samples] within a Case, or</li> <li>Each 7 calendar day period (3 calendar day period for 7 day turnaround) during which field samples in a Case are received (said period beginning with the receipt of the first sample in the SDG).</li> </ul> <p>In addition, all samples and/or sample fractions assigned to an SDG must have been scheduled under the same contractual turnaround time. Preliminary Results have <b>no impact</b> on defining the SDG.</p>
<p>Exhibit B: Section 1.1 Footnote 2</p>	<p>A Sample Delivery Group (SDG) is a group of samples within a Case, received over a period of 7 calendar days (3 calendar day period for 7 day turnaround) or less and not exceeding 20 samples (excluding PE samples). Data for all samples in the SDG are due concurrently. The date of delivery of the SDG or any samples within the SDG is the date that the last sample in the SDG is received. See Exhibit A for further description.</p>

Exhibit Section(s)	Revisions
Exhibit B: Section 1.1 Table 1	<p>The delivery schedule for Preliminary Results has been changed as follows:</p> <ul style="list-style-type: none"> <li>• VOA Analyses - Within 48 hours after receipt of each sample at laboratory, if requested.</li> <li>• SV and Pest Analyses - Within 72 hours after receipt of each sample at laboratory, if requested.</li> </ul>
Exhibit B: Section 2.2	<p>If submitted documentation does not conform to the criteria as specified in the contract, the Contractor is required to resubmit the documentation with deficiency(ies) corrected. This documentation shall be submitted within six (6) business days at no additional cost to the Government.</p>
Exhibit B: Section 2.2.1	<p>The Contractor shall respond within seven (7) days to written requests from Regional data recipients for additional information or explanations that resulted from the Governments's inspection activities unless otherwise specified in the contract.</p>
Exhibit B: Section 2.6	<p>The Contractor shall retain a legible electronic (PDF) or hard copy of the Complete SDG File (not the Sample Data Package) for 365 days after submission of the reconciled data package. After this time, the contractor may dispose of the package.</p>
Exhibit B: 3.3.6	<p>The "SDG No." field is for the Sample Delivery Group (SDG) number. The SDG number is the EPA sample number of the first field sample received in the SDG, except when this would cause duplication. When several samples are received together in the first SDG shipment, the SDG number shall be the lowest sample number (considering both alpha and numeric designations) in the first group of samples received under the SDG. If fractions of the same field samples are scheduled under different turnaround times, thus creating separate SDGs containing the same sample numbers, a different sample number shall be utilized in the assignment of the SDG number for each SDG. If a situation arises where there are an insufficient number of samples for assignment of SDG numbers (i.e., 1 sample with a 14 day turnaround and 10 samples for a 21 day turnaround), the Contractor shall contact SMO for the assignment of an SDG number.</p>



Exhibit Section(s)	Revisions
Exhibit C: Section 1.0 Volatile Target Compound List and Contract Required Quantitation Limits	All values under the column "Med. Soil ug/kg" have been modified from 1200 to 1300.
Exhibit D/VOA Sections 8.1.4 and 10.1.5.3	The 4 grams sample size is to be used when performing the medium level preparation <u>in the laboratory</u> . For example, the laboratory has received a container of soil which is to be extracted with methanol in the laboratory. The 5 gram sample size is to be used when soil samples are preserved with methanol <u>in the field</u> . The Medium Soil Quantitation Limits in Section 1.0 of Exhibit C are based upon the "in laboratory" protocol using 4 grams of sample. Even though the "in field" protocol would present a lower quantitation limit (1000 ug/Kg), the required quantitation limit will remain at 1300 ug/Kg)
Exhibit D/VOA Section 10.1.3.7	This section stipulates that 10 uL of system monitoring compound spiking solution and 10 uL of internal standard spiking solution shall be added to each 5 mL water sample analyzed. This requirement is based on a manually operated purge and trap instrumentation. Purge and trap instrumentation is widely available in which internal standards and system monitoring compounds are automatically added to each sample. Some of this instrumentation may be set-up by the manufacturer to add only 1 uL of internal standards or system monitoring compounds. The 1 uL addition of standards will be allowed if the addition is done solely in an automated manner, and if the final concentration of standard in the 5 mL water sample remains the same as specified in the current SOW (i.e., 50 ug/L internal standard analyte and system monitoring compound concentration).
Exhibit D/VOA Section 10.1.4.1	The following language is added to this section:  If the Agency specifically requests the laboratory to analyze a sample only by the medium level protocol (i.e., methanol extraction technique), the laboratory is not obligated to perform the low level analysis. This request will be specified on the Traffic Report. If the laboratory receives a Traffic Report specifying such request, the laboratory is to confirm the request through SMO prior to proceeding with sample analysis.

Exhibit Section(s)	Revisions
Exhibit D/VOA Appendix B Section 9.4.1	Prior to sample purge, all soil/sediment samples must be allowed to warm to ambient temperature. For those samples that have been stored in freezing compartments and that will be analyzed by the low concentration level protocol, 5 mL of reagent water must be added to the vials without distributing the hermetic seal of the sample vial (Note: an additional 5mL of reagent water will be added to the vial as per section 9.4.2). Shake all vials containing aqueous solutions gently, to ensure that the contents move freely and that stirring will be effective. Place the sample vial in the instrument carousel according to the manufacturer's instruction.
Exhibit G:	<p>The following definitions have been added:</p> <ul style="list-style-type: none"> <li>Contract Compliance Screening (CCS) - a screening of diskette and hardcopy data deliverables for completeness and compliance with the contract. This screening is done under USEPA direction by the SMO contractor.</li> <li>Contract Laboratory Program (CLP) - supports USEPA's Superfund effort by providing a range of state-of-the-art chemical analytical services of known quality. This program is directed by the Analytical Operations/Data Quality Center of USEPA OERR.</li> <li>USEPA Regional CLP Project Officer - the Regional USEPA official responsible for monitoring laboratory performance and/or requesting analytical data or services from a CLP laboratory.</li> <li>USEPA AOC Organic Program Manager - the USEPA Analytical Operations/Data Quality Center (AOC) official who manages the CLP Organic program.</li> </ul>